P⁎TENT COOPERATION TREA⁓⁄

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents
United States Patent and Trademark Office
Box PCT
Washington, D.C.20231
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year)
28 August 2000 (28.08.00)

International application No.	Applicant's or agent's file reference
PCT/SE99/02392	43999-56510

International filing date (day/month/year)	Priority date (day/month/year)
16 December 1999 (16.12.99)	18 December 1998 (18.12.98)

Applicant
ANDERSSON, Conny et al

1. The designated Office is hereby notified of its election made:

    [X] in the demand filed with the International Preliminary Examining Authority on:

    28 June 2000 (28.06.00)

    [ ] in a notice effecting later election filed with the International Bureau on:

2. The election [X] was

    [ ] was not

    made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland	Authorized officer Nestor Santesso
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

Form PCT/IB/331 (July 1992)

SE9902392

TENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 43999-56510	**FOR FURTHER ACTION**	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No. PCT/SE99/02392	International filing date *(day/month/year)* 16.12.1999	Priority date *(day/month/year)* 18.12.1998
International Patent Classification (IPC) or national classification and IPC7 C22C 33/08 // G01N 25/02		
Applicant SinterCast AB et al		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of ___4___ sheets, including this cover sheet.

   ☐ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of _____ sheets.

3. This report contains indications relating to the following items:

   I ☒ Basis of the report

   II ☐ Priority

   III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability

   IV ☐ Lack of unity of invention

   V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

   VI ☐ Certain documents cited

   VII ☐ Certain defects in the international application

   VIII ☐ Certain observations on the international application

Date of submission of the demand 28.06.2000 -	Date of completion of this report 03.05.2001
Name and mailing address of the IPEA/SE Patent- och registreringsverket Telex Box 5055 17978 S-102 42 STOCKHOLM PATOREG-S Facsimile No. 08-667 72 88	Authorized officer Anna-Maj Magnusson/MP Telephone No. 08-782 25 00

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

| I. | **Basis of the report** |

1. With regard to the **elements** of the international application:*

☒ the international application as originally filed

☐ the description:

pages _____ , as originally filed

pages _____ , filed with the demand

pages _____ , filed with the letter of _____

☐ the claims:

pages _____ , as originally filed

pages _____ , as amended (together with any statement) under article 19

pages _____ , filed with the demand

pages _____ , filed with the letter of _____

☐ the drawings:

pages _____ , as originally filed

pages _____ , filed with the demand

pages _____ , filed with the letter of _____

☐ the sequence listing part of the description:

pages _____ , as originally filed

pages _____ , filed with the demand

pages _____ , filed with the letter of _____

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language _____ which is:

☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).

☐ the language of publication of the international application (under Rule 48.3(b)).

☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/ or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. ☐ The amendments have resulted in the cancellation of:

☐ the description, pages _____

☐ the claims, Nos. _____

☐ the drawings, sheet/fig _____

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2 (c)).**

* *Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).*

** *Any replacement sheet containing such amendments must be referred to under item I and annexed to this report.*

Form PCT/IPEA/409 (Box I) (January 1998)

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims	1-6		YES
	Claims			NO
Inventive step (IS)	Claims	1-6		YES
	Claims			NO
Industrial applicability (IA)	Claims	1-6		YES
	Claims			NO

2. Citations and explanations (Rule 70.7)

The claimed invention relates to a process and an apparatus for determining the amount of structure-modifying agent that has to be added to a cast iron melt to obtain either compacted or spheroidal graphite cast iron. The apparatus also comprises a computer program.

The invention further relates to a process for producing cast iron in which the above process is employed.

The following documents are cited in the International Search Report:

D1: WO 8601755 A1

D2: WO 9113176 A1

D3: WO 9206809 A1

All the documents are cited as background art.

D1 is considered to be the most relevant document. D1 discloses a method for producing compacted graphite cast iron by using thermal analysis. A sample is taken from a bath of molten cast iron and cooling curves are recorded for this sample. It is then possible to determine the amount of structure-modifying agents that must be added to the melt to obtain the desired microstructure. (Cf. abstract.)

However, the method according to D1 can not be used when the molten cast iron solidifies as eutectic or hypereutectic iron.

.../...

Supplemental Box
(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: V.

The claimed invention differs from this previously known
technology in that it provides a possibility to evaluate
cooling curves recorded in near-eutectic cast iron melts. This
evaluation is then used to determine the amount of structure-
modifying agents that must be added to the melt to obtain the
desired microstructure.

There is nothing in D1 that would lead a person skilled in the
art to modify that method so as to arrive at the one described
in the claims.

Thus, the claimed invention is novel and is considered to
include an inventive step. It is also considered to fulfil the
criteria of industrial applicability.

(54) Title: METHOD FOR DETERMINING THE NEEDED AMOUNT OF STRUCTURE MODIFYING AGENT TO BE ADDED TO CAST IRON

(57) Abstract

     The present invention provides a possibility to evaluate cooling curves recorded in near–eutectic cast iron melts. The curves are evaluated by determining the net amount of heat generated in the melt in the centre of the sample to the melt as a function of time. This information is then used to identify the part of the centrally recorded cooling curve that can be used as a basis for determining the amount of structure–modifying agent that must be added to produce compacted graphite cast iron, and/or spheroidal graphite cast iron, and to identify the part of said curve that is associated with formation of primary austenite.

METHOD FOR DETERMINING THE NEEDED AMOUNT OF STRUCTURE MODIFYING AGENT TO BE ADDED TO CAST IRON

The present invention relates to an improved method for predicting the microstructure in which a cast iron melt, having a composition with a carbon equivalent near

5   the eutectic point of the iron-carbon phase diagram, will solidify. The invention also relates to an apparatus for carrying out the method.

Background of the invention

10   WO86/01755 (incorporated by reference) discloses a method for producing compacted graphite cast iron by using thermal analysis. A sample is taken from a bath of molten cast iron and this sample is permitted to solidify during 0.5 to 10 minutes. The temperature is recorded simultaneously by two temperature-responsive means, one of which is arranged in the centre of the sample and the other in the immediate

15   vicinity of the vessel wall. So-called cooling curves representing temperature of the iron sample as a function of time are recorded for each of the two temperature-responsive means. According to this document, it is then possible to determine the necessary amount of structure-modifying agents that must be added to the melt in order to obtain the desired microstructure. However, the cooling curves disclosed in

20   this document are rather uniform and no variations are disclosed.

In order to accurately determine the graphite microstructure in cast iron specimens, conventional thermal analysis techniques, such as the one disclosed in WO 86/01755, require cooling curves where the first thermal arrest caused by austenite

25   formation is distinctly separated from heat release caused by the onset of eutectic solidification. However, sometimes cooling curves are obtained without such a distinctly separated thermal arrest. This is the case when the molten cast iron solidifies as eutectic or hyper-eutectic iron. Until now, it has not been possible to use cooling curves corresponding to near eutectic cast iron for monitoring graphite growth.

30

WO93/20965 teaches that hyper-eutectic melts, where graphite nucleates before iron, do not provide a distinct plateau as the temperature crosses the liquidus line. This is correctly attributed to the fact that graphite crystallisation has a lower latent heat release than iron.

5   By placing a small amount of low-carbon iron in the melt, the low-carbon iron partially dissolves locally while the sample is still molten. As the sample cools, the relatively pure iron surrounding the remaining solid portion of the nail begins to solidify because of its lower carbon equivalent (CE). Ultimately, as the sample volume

10   ume cools below the liquidus line, the remaining solid volume of nail, the surrounding low CE and the melted portion of the nail begin to solidify and "trigger" the solidification in an otherwise hyper-eutectic melt. The net result is that an austenite arrest plateau appears on the cooling curve.

15   WO93/20965 also states that the temperature difference (ΔT) between T_δ and $T_{c\,max}$ can be used to establish a correlation with the carbon equivalent. However, WO93/20965 refers to hyper-eutectic melts, i.e. melts where the carbon equivalent is so high that the release of heat from the primary solidification does not coincide with the minima of the cooling curve (in the hatched region of Fig. 2(a) of

20   WO93/20965). Accordingly, the primary solidification and the eutectic solidification are separate.

None of the above cited references discuss anything about carrying out thermal analysis on cast iron melts in order to determine the carbon equivalent of melts

25   which are near-eutectic. Moreover, WO93/20965 suggests measurements on melts having a carbon equivalent of up to 4.7%. It is disadvantageous to reach such high values because of graphite flotation and the degeneration of graphite shape. Likewise, the method of WO93/20965 is disadvantageous in that it requires an extra addition of low-carbon steel or iron to the sampling vessel, and accordingly lead to

30   higher costs and a more laborious method.

Summary of the invention

The present invention provides a possibility to evaluate cooling curves recorded in near-eutectic cast iron melts. The curves are evaluated by determining the net amount of heat generated in the centre of the melt sample as a function of time. This information is then used to identify the part of the centrally recorded cooling curve that can be used as a basis for determining the amount of structure-modifying agent that must be added to produce compacted graphite cast iron, and/or spheroidal graphite cast iron, and to identify the part of said curve that is associated with formation of primary austenite.

Definitions

The term "cooling curve" as utilised herein refers to graphs representing temperature as a function of time, which graphs have been recorded in the manner disclosed in WO86/01755, WO92/06809.

The term "heat generation curve" as utilised herein relates to a graph showing the heat that is generated in a certain zone of a molten cast iron. For the purposes of the present invention, all heat generation curves herein are determined for a zone located in the centre of a sample of molten cast iron. This zone is generally referred to as the "A zone".

The term "sample vessel" as disclosed herein, refers to a small sample container which, when used for thermal analysis, is filled with a sample of molten metal. The temperature is then recorded during solidification in a suitable way. preferably the sample vessel is designed in the manner disclosed in WO86/01755, WO92/06809, WO91/13176 (incorporated by reference) and WO96/23206 (incorporated by reference).

The term "sampling device" as disclosed herein, refers to a device comprising a
sample vessel equipped with at least two temperature responsive means for thermal
analysis, said means being intended to be immersed in the solidifying metal sample
5   during analysis, and a means for filling the sample with molten metal. The sample
vessel is preferably equipped with said sensors in the manner disclosed in
WO96/23206.

The term "structure-modifying agent" as disclosed herein, relates to compounds af-
10 fecting the morphology of graphite present in the molten cast iron. Suitable com-
pounds can be chosen from the group of magnesium and rare earth metals such as
cerium, or mixtures of these compounds. The relationship between the concentra-
tion of structure-modifying agents in molten cast irons and the graphite morphology
of solidified cast irons have already been discussed in the above cited documents
15 WO92/06809 and WO86/01755.

The term "CGI" as utilised herein refers to compacted graphite cast iron.

The term "SGI" as utilised herein refers to spheroidal graphite cast iron.

20

Figures

The invention is described with reference to the accompanying figures, in which:

25 Figs. 1A-4A disclose cooling curves. A continuos line relates to the temperature in
the centre of the melt and a dashed line relates to the temperature close to the wall
of the sample vessel. Figs. 1B-4B show A zone heat generation curves correspond-
ing to the cooling curves in figs. 1A-4A. Fig. 1 relates to normal hypo-eutectic cast
iron and figs 2-3 show curves for near-eutectic cast irons comprising increased val-
30 ues of the carbon equivalent. Fig. 4 discloses curves relating to hypo-eutectic cast
iron.

Fig. 5 is a schematic presentation of an apparatus for controlling production of compacted graphite or spheroidal graphite cast iron according to the present invention.

5

Fig. 6 is a schematic representation of a sample vessel in which the heat is approximately uniformly transported in all directions. During measurements the vessel is filled with molten cast iron. This molten cast iron can be considered as a freezing sphere. The A zone relates to a sphere in the centre of the molten sample and the B

10   zone relates to the molten iron surrounding the A zone. The radii r_1 and r_2 relate to the mean radii of the A zone and the B zone, respectively.

Detailed description of the invention

15   As already mentioned above, the present invention relates to an improved method for interpreting cooling curves in near-eutectic cast iron melts. One of the important aspects of a cooling curve is the maximum slope of the recalescence peak of the centrally recorded cooling curve. This point is referred to as α in figures 1A, 2A and 3A. In cooling curves corresponding to near-eutectic cast iron melts, the inflexion

20   point (referred to in the figures as "t_p") is located much closer to α and it can be very difficult to determine the slope correctly. Examples of such curves can be found in figure 2A and 3A. However, it has now turned out that the determination of the maximum slope at α can be considerably simplified if a heat generation curve corresponding to the central zone (the A zone) of the molten cast iron is calculated.

25   Such a heat generation curve renders it possible to determine the location of t_p in the centrally recorded cooling curve and to check whether it affects the slope of the recalescence peak or not.

The thermal balance of any uniform element can be described by the relation:

30

$$Q_{stored} = Q_{generated} + Q_{in} - Q_{out} \tag{1}$$

where Q_{stored} is the amount of heat stored by the heat capacity of the material, $Q_{generated}$ is the amount of heat generated by the volume of material, Q_{in} is the heat

5   transferred into the material from its surroundings and Q_{out} is the heat transferred out of the material to its surroundings.

When carrying out the present invention, it is advantageous to use a sample vessel as disclosed in SE 9704411-9. In such a sample vessel, the heat transport in a sam-

10   ple contained in the vessel is approximately the same in all directions. From now on the heat transport between the centre (Fig. 6, zone A) and the more peripheral (Fig. 6, zone B) part of a molten cast iron contained in a sample vessel is described. As the A zone is situated in the centre of the molten cast iron sample, and as that zone can be considered to be a freezing sphere, no heat is transported into the zone

15   and Q_{in} is therefore equal to zero. Appropriate substitutions in relation (1) above give the following equation:

$$C_p m_A \, dT_A/dt = Q_{genA} + 0 - 4\pi ke \left[(T_A - T_B)/(1/r_1 - 1/r_2) \right] \tag{2}$$

20   where C_p is the heat capacity per unit mass, m_A is the mass of the A zone, dT_A/dt is the temperature change of the A zone per unit time, Q_{genA} is the heat generated in zone A, ke is the effective heat transfer coefficient of the material, and $(1/r_1 - 1/r_2)^{-1}$ is the mean distance for heat transport. The radii r_1 and r_2 are both defined in fig. 6. T_A and T_B are the temperatures in the A zone and B zone respectively.

25

From equation (2) we can isolate the heat generation term and calculate the average bulk heat generation in the A zone:

$$Q_{genA} = C_p m_A dTA/dt + 4\pi ke \left[(T_A - T_B)/(1/r_1 - 1/r_2) \right] \tag{3}$$

30

In equation (3) all variables are constants except dT_A/dt and $(T_A - T_B)$. Accordingly, equation (3) can be simplified to:

$$Q_{genA} = k_1 \, dT_A/dt + k_2(T_A - T_B) \tag{4}$$

5

where k_1 and k_2 are constants. Hence, a heat release curve can be calculated from a set of cooling curves recorded in the centre and the periphery of a sample of molten cast iron.

10 As already mentioned, these calculations are based on a situation where heat is uniformly transported in all directions. The skilled person can of course also calculate other equations corresponding to other heat transport conditions.

As mentioned earlier, the carbon equivalent of a certain cast iron melt affects the
15 appearance of its corresponding cooling curves and heat release curves. Such cooling curves and heat generation curves can, depending on the carbon equivalent, be classified into one of four model types:

Type 1, which is disclosed in fig. 1, relates to hypo-eutectic cast iron. The cooling
20 curve recorded in the centre of the sample is characterised by a first inflexion point associated with formation of austenite, followed by a local minimum. Interpretation of such curves does not present any particular difficulties and can be carried out in accordance with the principles outlined in WO86/01755 and WO92/06809. It should be noted that there is a peak (t_p) on the zone A heat generation curve that
25 corresponds to the inflexion point (and accordingly with the onset of austenite formation) on the centrally recorded cooling curve.

Type 2, which is disclosed in fig. 2, relates to near-eutectic cast iron and is characterised by a very flat local minimum on the cooling curve recorded in the centre of
30 the sample. An inflexion point cannot be seen. However, it is much easier to find a peak corresponding to the austenite formation point t_p on the heat generation curve.

In this case, it turns out that the austenite formation point t_p is located in connection to the local minimum resulting in a very flat minimum. From figures 2A and 2B it can be seen that t_p does not affect the slope at α.

5   Type 3, which is disclosed in fig. 3, also relates to near-eutectic cast iron, and is characterised by a detectable inflexion point after the local minimum in the cooling curve recorded in the centre of the sample. After determining the heat generation curve it is easy to establish the position of the austenite formation point t_p to a location immediately after the local minimum. However, in this situation the recales-

10   cence, and accordingly α is affected by the austenite formation. In order to be able to disregard the effects of the "moved austenite formation point" and to find a correct α located at t_α (where $t_\alpha > t_p$), the part of the centrally recorded cooling curve immediately adjacent to t_p, and having time values t such that $t - t_p$ are larger than a predetermined threshold value tv, are searched for a maximum value α_1 of the first

15   time derivative. This value α_1 is then used as a correct α in the method. A suitable threshold value tv can easily be determined by the skilled person, but typical examples of such values are 1-5 seconds.

Type 4, which is disclosed in fig. 4A, relates to hyper-eutectic cast iron. This type is

20   characterised in that no evident inflexion points or wide minima can be seen. The heat generation curve of the A zone does not comprise any detectable peaks. The present invention is not applicable in this situation.

It is preferred to carry out the prediction method by using a computer-controlled

25   system, especially when a large number of measurements must be carried out. In this case, the same kind of sampling device 22 that has been described above is used. Such a computer-controlled system is outlined in fig. 6. During the measurement of a particular sample the two temperature-responsive means 10, 12 send signals to a computer 14 comprising a ROM unit 16 and a RAM unit 15 in order to

30   generate the cooling curves. The computer device 14 is coupled to a memory means

19 in which a program routine is stored. When, in the following, it is described that the computer operates, it is to be understood that the computer is directed by the program in memory 19. The memory 19 may be a ROM circuit or a hard disc. The program may be supplied to the memory 19 from a non-volatile memory such as a

5   CD-ROM or a diskette, e.g. via a databus (not shown). The computer has access to calibration data in a ROM unit 16 and calculates the amount of structure-modifying agents that must be added to the melt. This amount is signalled to a means 18 for administrating structure-modifying agent to the melt 20 to be corrected, whereby the melt is supplied with an appropriate amount of such agents.

10

Claims

1. A process for determining the amount of structure-modifying agent that has to be added to a certain cast iron melt in order to obtain compacted graphite cast iron or spheroidal graphite cast iron from a molten cast iron having an hypo-eutectic or close to eutectic composition, which method requires a sampling device comprising a sample vessel, means for monitoring temperature as a function of time both in the centre and close to the wall of said sample vessel, and a means for administrating structure-modifying agents to the molten cast iron, the method comprising the steps of:

a) for the chosen casting method calibrating the amount of structure-modifying agent that has to be added to a hypo-eutectic melt in order to obtain compacted graphite cast iron or spheroidal graphite cast iron as a function of the maximum value α of the first time derivative of a cooling curve recorded in the centre of a sample vessel;

b) taking a sample of the molten cast iron by using a sampling device;

c) allowing said sample to solidify in a sample vessel, and during the solidification recording cooling curves in the centre of the sample vessel and at the sample vessel wall, respectively;

d) determining a heat generation curve disclosing the heat generated in the centre of the sample as a function of time by applying

   i) the thermal balance formula:

   $$Q_{stored} = Q_{generated} + Q_{in} - Q_{out}$$

   where Q_{stored} is the amount of heat stored by the heat capacity of the material, $Q_{generated}$ is the amount of heat generated by the volume of

material, Q_{in} is the heat transferred into the material from its surround-ings and Q_{out} is the heat transferred to the surroundings; and

ii) the cooling curves recorded in step c);

e) identifying the location of a possible local maximum t_p on the heat generation curve obtained in step d), which maximum corresponds to an austenite formation point on the centrally recorded cooling curve obtained in step c), and controlling whether there is a risk that this austenite formation point affects the value of the maximum slope of the centrally recorded cooling curve; and

f) if a t_p has been located, and if there is no risk that the maximum first time deriva-tive value α is affected by austenite formation, calculating the amount of struc-ture-modifying agent (Va) that has to be added to the melt by using the value α and the calibration data obtained in step a); or

g) if a t_p has been located, and if $t_p - t_\alpha$ is less than a threshold value t_{tv}, identifying a time, $t_{\alpha 1}$, $(t_{\alpha 1} > t_\alpha)$ for which the second time derivative of the centrally recorded cooling curve is approximately 0, determining the first time derivative value α_1, and calculating the amount of structure-modifying agent (Va) that has to be added to the melt by using the value α_1 and the calibration data obtained in step a).

2. A process according to claim 1, **characterised** in that the amount of structure-modifying agent that has to be added to a certain cast iron melt in order to obtain compacted graphite cast iron is determined.

3. A process according to claim 1 or claim 2, **characterised** in that the heat trans-port in a sample vessel containing molten cast iron is approximately the same in all directions.

4. A process for producing compacted graphite cast iron castings or spheroidal graphite cast iron castings from a molten cast iron having an eutectic or close to eutectic composition, comprising the steps of:

a) providing molten cast iron having an eutectic or close to eutectic composition;

5   b) determining the amount of structure-modifying agent that has to be added to a certain cast iron melt in order to obtain compacted graphite cast iron or spheroidal graphite cast iron according to the method of either claim 1 or claim 3;

c) adding the amount of structure-modifying agent determined in step b) to the molten cast iron; and

10   d) carrying out the casting operation in a per se known manner.

5. An apparatus for establishing, in real time, an amount of a structure-modifying agent to be added to a hypo-eutectic or near-eutectic cast iron melt during the process of producing a compacted graphite iron casting;

15   the apparatus comprising:

a first temperature sensor (10) for recording a cooling curve in the centre of a sample vessel;

a second temperature sensor (12) for recording a cooling curve in the vicinity

20   of the sample vessel wall;

a computer device (14) for determining an amount value (Va) of a structure modifying agent to be added to the melt;

a memory means (16) which is provided with pre-recorded calibration data;

the computer being set up to determine the heat generated in the centre of the

25   sample as a function of time (a heat generation curve) by applying

i) the thermal balance formula:

$$Q_{stored} = Q_{generated} + Q_{in} - Q_{out}$$

where Q_{stored} is the amount of heat stored by the heat capacity of the material, $Q_{generated}$ is the amount of heat generated by the volume of

30   material, Q_{in} is the heat transferred into the material from its surroundings and Q_{out} is the heat transferred to the surroundings; and

ii) the cooling curves recorded by said first and second temperature

sensors (10, 12);

the computer being set up to identify a time value t_p, corresponding to a local

5   maximum of the heat generated in the centre of the sample as a function of

time;

the computer being set up to calculate the maximum value of the first time de-

rivative of the cooling curve recorded by the first temperature sensor (10) and

to assign this value to the variable α and to assign the corresponding time to

10   the variable t_α;

the computer being set up to compare time values t_p and t_α, and

if $t_\alpha - t_p$ is less than a threshold value tv

the computer being set up to identify a new first derivative

value α, located at a time value $t_{\alpha 1}$, which is larger than t_α,

15   and which corresponds to a part of the cooling curve re-

corded by the first temperature sensor (10) where the sec-

ond time derivative is approximately 0 and to assign this

new first derivative value to the variable α;

the computer being set up to determine an amount value (Va) of a structure modi-

20   fying agent to be added to the melt by using the first derivative value α and pre-r-

ecorded calibration data.

6. A computer program product for use in an apparatus for establishing, in real time,

an amount of a structure-modifying agent to be added to a cast iron melt (20)

25   during the process of producing a compacted graphite iron casting;

wherein the apparatus has

.   a first temperature sensor (10) for recording a cooling curve in the cen-

tre of a sample vessel;

a second temperature sensor (12) for recording a cooling curve in the

30   vicinity of the sample vessel wall;

a computer device (14) for determining an amount value (Va) of a structure modifying agent to be added to the melt;

a memory means (16) which is provided with pre-recorded calibration data;

5   the computer program product comprising:

a recording medium and a computer-readable code means for directing the computer device to determine the heat generated in the centre of the sample as a function of time (a heat generation curve) by applying

i) the thermal balance formula:

$$Q_{stored} = Q_{generated} + Q_{in} - Q_{out} \quad .$$

10   where Q_{stored} is the amount of heat stored by the heat capacity of the material, $Q_{generated}$ is the amount of heat generated by the volume of material, Q_{in} is the heat transferred into the material from its surroundings and Q_{out} is the heat transferred to the surroundings; and

15   the cooling curves recorded by said first and second temperature sensors (10, 12);

a recording medium and a computer-readable code means for directing the computer device to identify a time value t_p, corresponding to a local maximum of the heat generated in the centre of the sample as a function of time;

20   a recording medium and a computer-readable code means for directing the computer device to calculate the maximum value of the first time derivative of the cooling curve recorded by the first temperature sensor (10) and to assign this value to the variable α and to assign the corresponding time to the variable t_α;

25   a recording medium and a computer-readable code means for directing the computer device to compare time values t_p and t_α, and

if $t_\alpha - t_p$ is less than a threshold value tv

to identify a new first derivative value α, located at a time value $t_{\alpha1}$, which is larger than t_α, and which corresponds

30   to a part of the cooling curve recorded by the first tem-

perature sensor (10) where the second time derivative is approximately 0 and to assign this new first derivative value to the variable α;

a recording medium and a computer-readable code means for directing the computer device to determine an amount value (Va) of a structure modifying agent to be added to the melt by using the first derivative value α and pre-recorded calibration data.
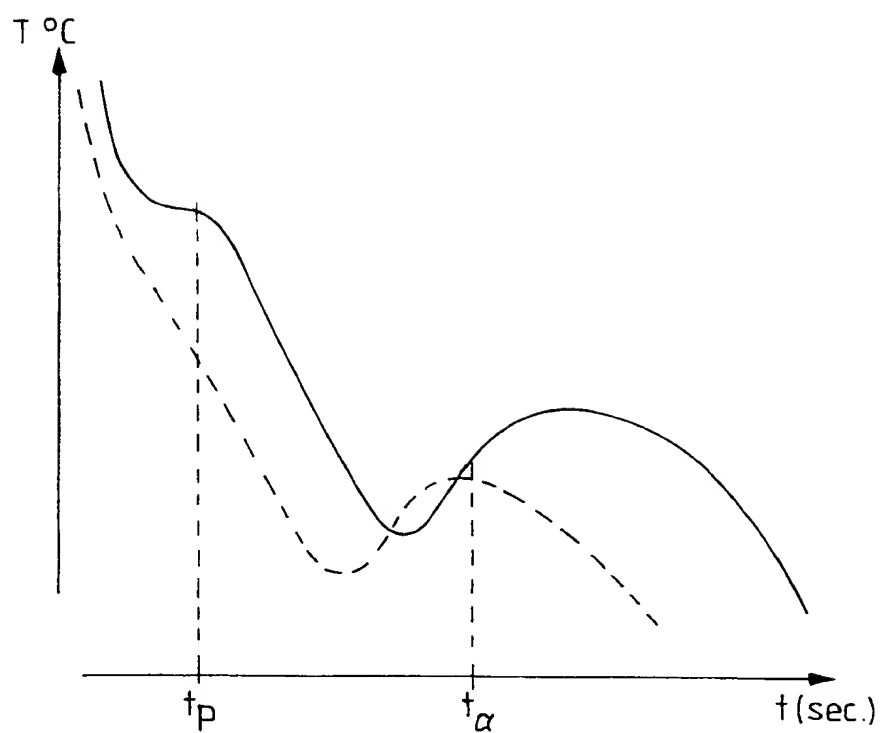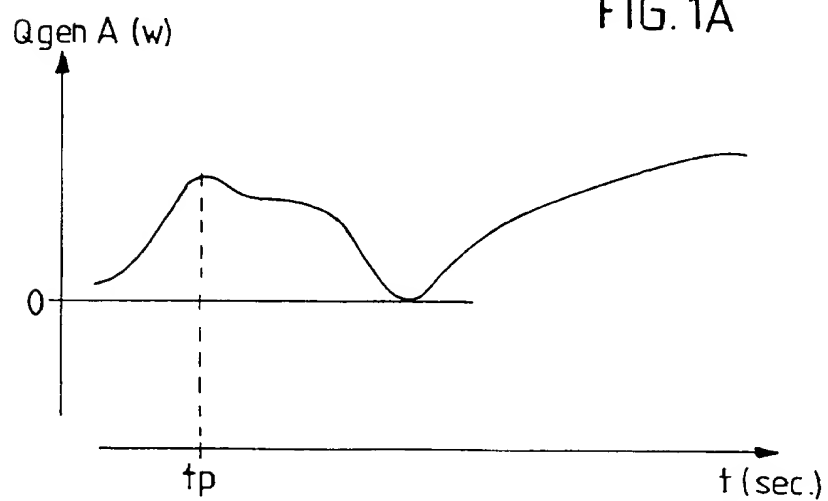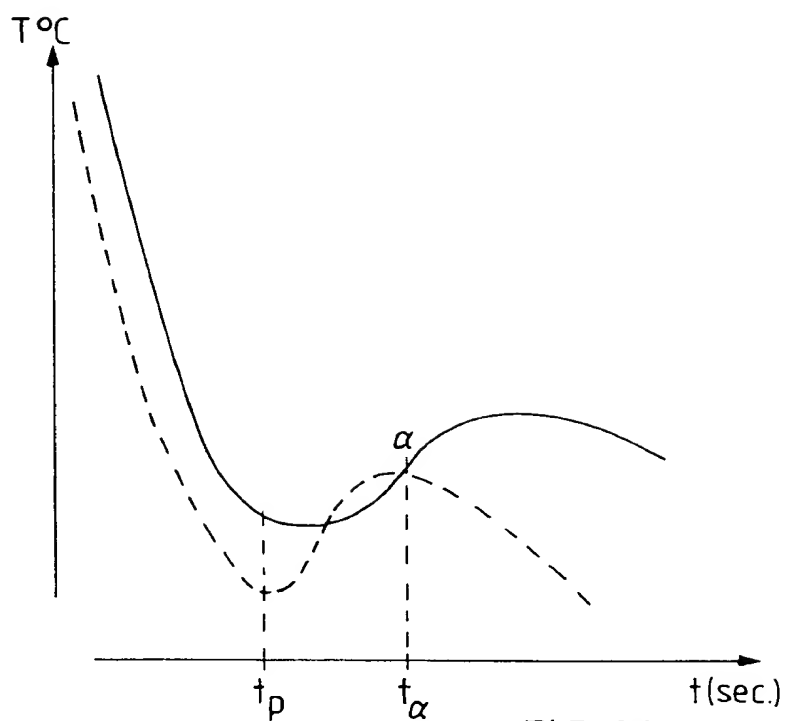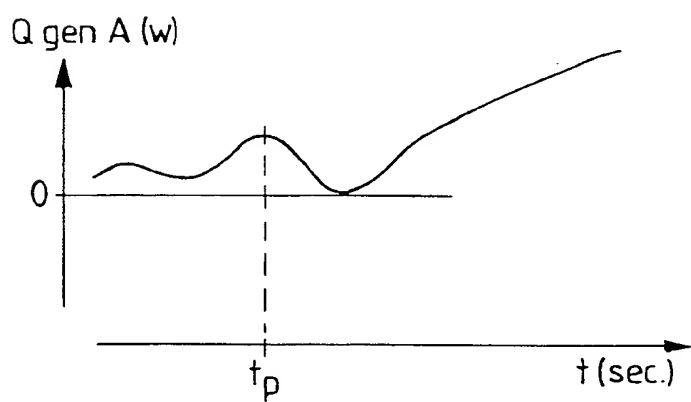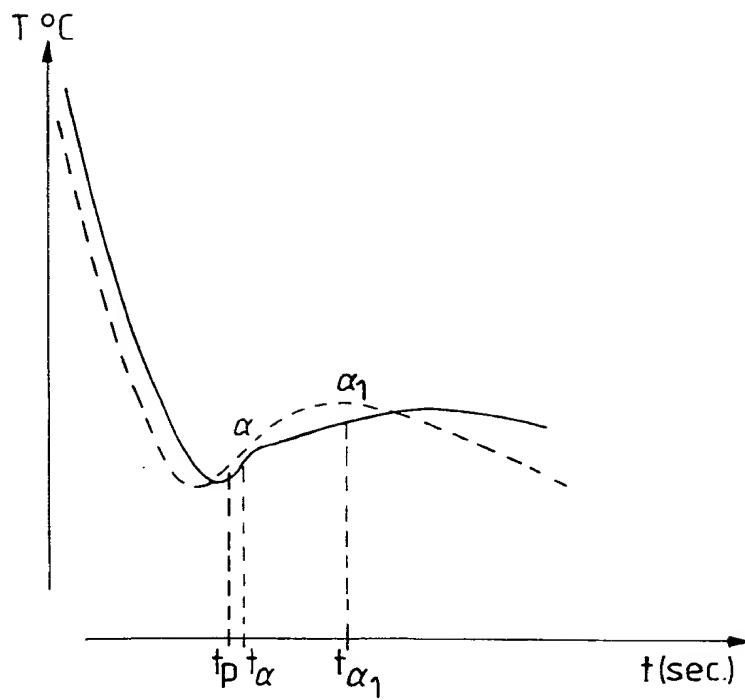
FIG.1A

FIG.1B

FIG. 2A

FIG. 2B

FIG. 3A

FIG. 3B

T°C

FIG.4A

Qgen A (w)

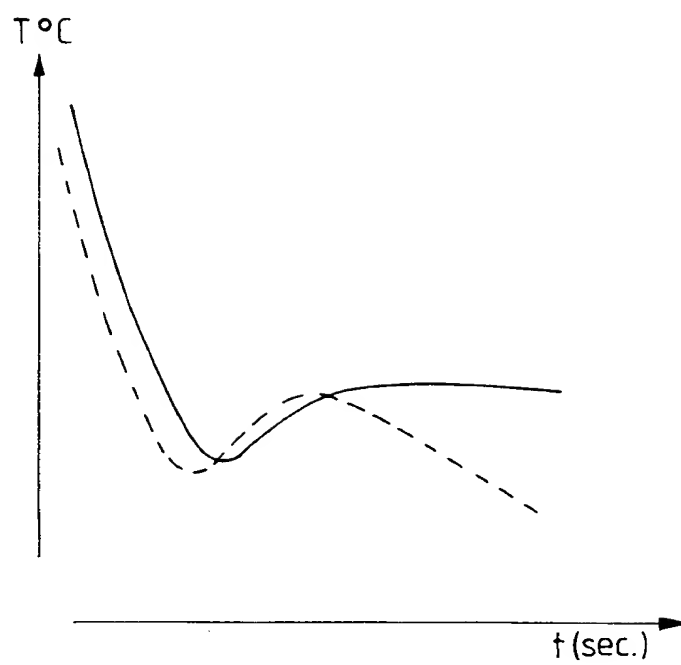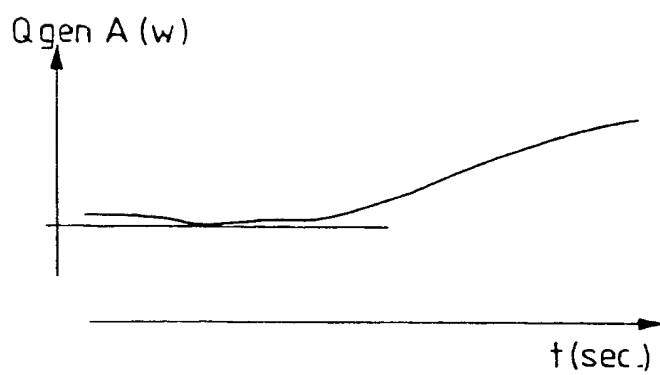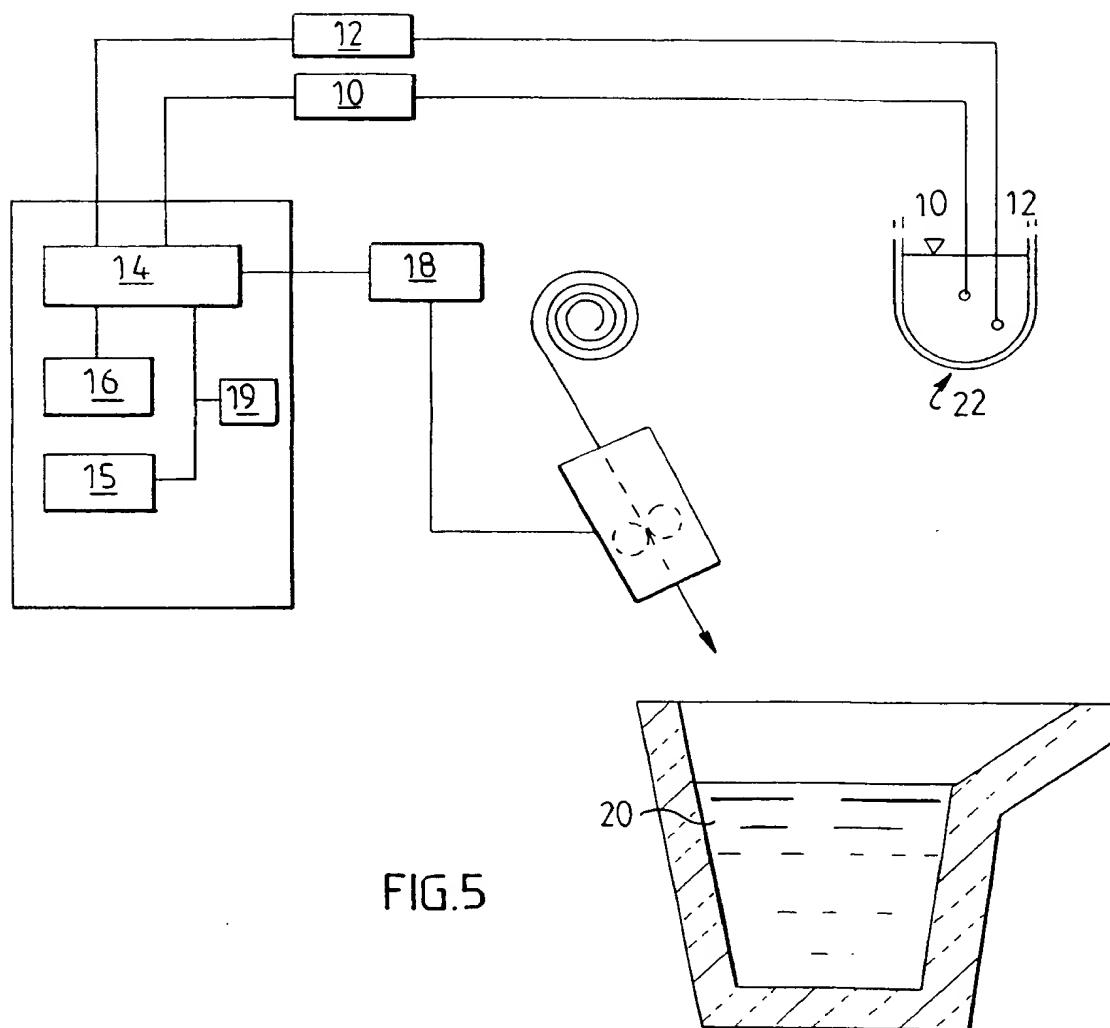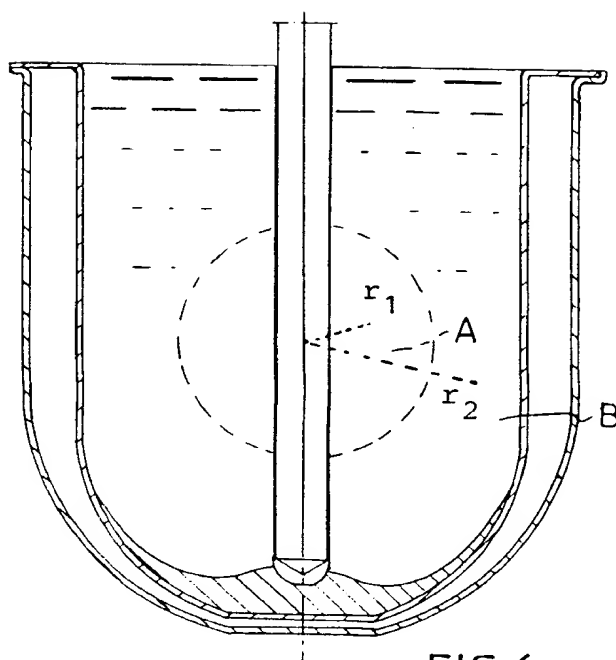
FIG.4B

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FIG.5

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FIG.6

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 99/02392

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: C22C 33/08 // G01N 25/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: C22C, G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	WO 8601755 A1 (SINTER-CAST AB), 27 March 1986 (27.03.86) --	1-6
A	WO 9206809 A1 (SINTER-CAST LTD.), 30 April 1992 (30.04.92) -- --------	1-6

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	
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"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	
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"P" document published prior to the international filing date but later than the priority date claimed	"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
29 March 2000	2 0 -04- 2000

Name and mailing address of the ISA/	Authorized officer
Swedish Patent Office Box 5055, S-102 42 STOCKHOLM	Anna-Maj Magnusson/MP
Facsimile No. +46 8 666 02 86	Telephone No. +46 8 782 25 00

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